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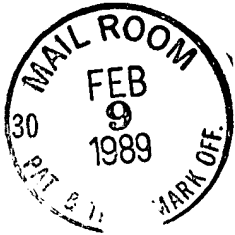
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FS(1343)BW

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: JEAN-YVES CHENARD ET AL : Group 143
U.S.Serial No. 254,313 :
Filed: April 15, 1981 :
For: IMPROVEMENT IN THE
STABILIZATION OF OF VINYL
HALIDE POLYMERS

AFFIDAVIT OF MICHEL FOURE
UNDER RULE 37 CFR 1.131

STATE OF NEW JERSEY)
)ss:
COUNTY OF MIDDLESEX)

MICHEL FOURE, being of legal age and under oath duly sworn, says and deposes:

That he is a chemist and has been working in the field of stabilizers for halogen containing polymers for five (5) years and has read and understands the above-identified patent application;

That prior to December 12, 1980, the following experiments were conducted in the United States on behalf of the Applicants:

EXPERIMENT

Four stabilizer compositions were prepared, each containing a mono-organo derivative of tetravalent tin where the remaining valences of the tin atom are satisfied by bonds to halogen or to halogen and sulfur. These compositions also contained a mercaptoloweralkanol ester of a carboxylic acid. Each of the four stabilizer compositions was evaluated as a stabilizer for vinyl halide polymers; a commercially available stabilizer was used as a control.

Stabilizer A

	<u>Parts by Weight</u>
Monobutyltin(isooctylmercaptoacetate)sulfide	1.82
$C_4H_9SnCl_3$	0.30
Mercaptoethyl oleate	4.93
Antioxidant ⁽¹⁾	0.25
Dioctyl phthalate	0.88

Stabilizer B

	<u>Parts by Weight</u>
$C_4H_9Sn(S)Cl$	0.93
$C_4H_9SnCl_3$	0.30
Mercaptoethyl oleate	4.93
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.78
Dioctyl phthalate	1.77

Stabilizer C

	<u>Parts by Weight</u>
Monobutyltin(isooctylmercaptoacetate)sulfide	2.28
$C_4H_9SnCl_3$	0.31
Mercaptoethyl oleate	3.71
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.28
Mineral oil ("Escoflex" 998) ⁽³⁾	1.30

Stabilizer D

	<u>Parts by Weight</u>
$C_4H_9Sn(S)Cl$	1.17
$C_4H_9SnCl_3$	0.31
Mercaptoethyl oleate	3.71
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.28
"Escoflex" 998 ⁽³⁾	2.41

(1) Available under trademark CAO-3 from Scherex Corp.

(2) Available from Sun Oil Company.

(3) Available from East Coast Chemicals.

The four stabilizer compositions described above, together with a commercially available stabilizer sold by M&T Chemicals Inc. under the trademark "THERMOLITE 310" were evaluated in a polyvinyl chloride formulation having the following composition:

	<u>Parts by Weight</u>
Vinyl chloride homopolymer ("Tenneco" 225PG) ⁽⁴⁾	100.00
Calcium carbonate ("Hydrocarb 30T") ⁽⁵⁾	3.0
Titanium dioxide	1.0
Calcium stearate	0.6
Paraffin wax ("XL 165") ⁽⁶⁾	1.0
Stabilizer	0.4

(4) Available from Tenneco Chemicals.

(5) Available from Omya Inc.

(6) Available from American Hoechst.

The formulations were evaluated using the procedure described in Example XIV of the above-identified application by fabricating sheets using a roll mill heated to a

temperature of 350°F. The sheets were removed from the mill after five minutes and then compression molded for ten (10) minutes at 350°F. Readings of the Whiteness Index (W.I.) and Yellowness Index (Y.I.) were directly obtained from a Macbeth MC 1500 colormeter.

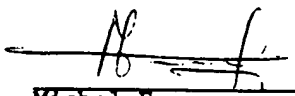
The results of the experiment are summarized below:

<u>Stabilizer</u>	<u>W.I.</u>	<u>Y.I.</u>
A	53.9	9.7
B	57.3	8.6
C	53.0	10.0
D	55.7	9.7
T-310 (control)	36.7	15.8

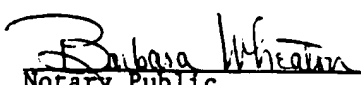
The experimental results indicate that each of the four stabilizer compositions is more effective to stabilize vinyl halide polymers with respect to heat than a commercially available stabilizer; each of the four stabilizer compositions results in test specimens displaying both increased Whiteness Index and decreased Yellowness Index, compared to a commercial stabilizer.

The experiments described above were performed in the United States prior to December 12, 1980, were performed by affiant or under his direction on behalf of the Applicants, and were contemporaneously recorded by affiant in his own hand in laboratory notebook 4384 page 36, a true copy of which (dates and certain extraneous matter having been deleted) is annexed hereto as Exhibit "A".

12-19-1983
Date


Michel Foure

Sworn to and subscribed before me on the date aforesaid by Michel Foure.


Notary Public

BARBARA WHEATON
NOTARY PUBLIC OF NEW JERSEY
My Commission Expires March 22, 1987

Form Page No. ...

"Bu-Sn^S-Ce" will be evaluated against CN 8900 ("Bu-Sn^S-IOMA" in "B+" and "C+" formulations using the same stoichiometry for tin -

Following stabilizers are prepared:

	39-A	39-B	39-C	39-D	
CN 8900	1.82	—	2.28	—	(BWRHL-81K
Bu Sn (S) Ce	—	0.93	—	1.17	(3763-49)
Bu Sn Cl ₃	0.30	0.30	0.31	0.31	
MEO L	4.93	4.93	3.71	3.71	(4342-04)
Carbocation M.O.	1.82	—	—	—	
CAO-3	0.25	0.12	0.12	0.12	
Super Oil 150	—	2.28	2.28	2.28	
Escoflex 998	—	1.30	1.30	2.41	
DOP	0.88	1.77	—	—	

* Results of press test (10 min, 350°F)

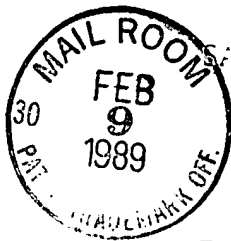
Test #	WI	YI	Stabilizers	
(4384)				
39-1	53.9	9.7	0.4	36-A
39-2	57.3	8.6	0.4	36-B
39-3	53.0	10.0	0.4	36-C
39-4	55.7	9.2	0.4	36-D
39-5	36.7	15.8	0.4	T310

← with Bu-Sn^S-Ce

Bu-Sn^S-Ce seems to improve the WI.

To Page No. 39

Understood by	Date	Invented by	File
M. Cresto		Michael Fourn	
		Recorded by	



SM(1343)K

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of

Applicant: JEAN-YVES CHENARD ET AL : Group 153

Serial No.: 254,313 : Examiner:
V. Hoke

Filed: April 15, 1981 :

For: IMPROVEMENT IN THE STABILIZATION
OF VINYL HALIDE POLYMERS

SUPPLEMENTAL DECLARATION OF M. FOURE -- 37 CFR 1.131

Hon. Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

I, MICHEL FOURE, do hereby declare that:

1. I am a Doctor Rerum Natur in Chemistry; this degree was conferred on me by the State University of Wurzburg (West Germany) in 1975; I did post-doctorate research in chemistry at the University of Osaka (Japan) from 1975-1977.
2. I was employed at the Lacq, France, Research Centre of Societe Nationale Elf Aquitaine from 1977 to 1980, doing research in cosmetics and fragrances, lubricants, process development, organotin biocides and polyvinyl chloride stabilizers; in 1980 I became employed by M&T Chemicals Inc., at Rahway, New Jersey, as a Research Associate in the Polyvinyl Chloride (PVC) Additives Group; in 1981 I was promoted to the position of Manager, PVC Additives Group; I hold five patents in the field of stabilization of PVC; I am familiar with the literature and state of the art related to the stabilization of PVC.

3. I am intimately familiar with the invention of Chenard, et al relating to the use of certain mercaptoesters in conjunction with an organotin stabilizer to stabilize polyvinyl chloride with respect to heat.
4. My Affidavit under 37 CFR 1.131, signed on December 19, 1983 is hereby incorporated by reference.
5. It was my opinion, when I first read the Chenard et al, French Patent No. 2,434,835 that a reasonably skilled chemist familiar with the art and literature related to stabilization of PVC would know that a variety of organotin compounds in addition to the compounds specifically named could successfully be used in combination with the described mercaptoesters; I continue to be of that opinion.
6. It was my belief that halogen-containing organotin compounds could successfully be used in combination with the described mercaptoesters; my opinion in this respect was based on my familiarity with the work of Chenard et al and with the prior art, which includes patents such as:

Molt.....	U.S. Patent.....	3,970,678
Wowk.....	U.S. Patent.....	3,758,537
Wowk.....	U.S. Patent.....	3,758,536
Wowk.....	U.S. Patent.....	3,758,341
Larkin.....	U.S. Patent.....	3,715,333
Wowk.....	U.S. Patent.....	3,665,025
Wowk.....	U.S. Patent.....	3,655,613
Kugele.....	U.S. Patent.....	4,118,371
Kugele.....	U.S. Patent.....	4,062,881
Kugele.....	U.S. Patent.....	3,979,359
Bakassian.....	U.S. Patent.....	3,943,099
Brecker.....	U.S. Patent.....	3,630,992
Albright & Wilson..	British Patent..	1,117,652

7. My belief that halogenated organotin compounds could be used in combination with mercaptoesters to obtain improved performance was confirmed by the experimental work described in my Affidavit of December 19, 1983, and it was clear, prior to December 12, 1980, that halogen-containing organotin compounds, in combination with mercaptoesters, gave improved results in respect of stabilization of PVC and that halogen-containing organotin compounds which were themselves known to be useful PVC stabilizers, could be used in combination with the mercaptoesters to stabilize PVC.

7-13-1984

Date



Michel Foure

Sworn to and subscribed before me on the date
aforesaid by Michel Foure.


Notary Public

BARBARA WHEATON
NOTARY PUBLIC OF NEW JERSEY
My Commission Expires March 28, 1987